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MEMORANDUM FOR PRS (Contractor/In-House Publication)

FROM: PROI (TI) (STINFO)

25 August 1999

SUBJECT: Authorization for Release of Technical Information, Control Number: AFRL-PR-ED-TP-FY99-0173
Wapner and Hoffman, "Utilization of Surface Tension and Wettability in the Design and Operation of

Microsensors"

Peer Review Journal Article

<u>UTILIZATION OF SURFACE TENSION AND WETTABILITY IN THE DESIGN</u> <u>AND OPERATION OF MICROSENSORS</u>

Phillip G. Wapner and Wesley P. Hoffman

Abstract

The behavior of fluid droplets contained within shaped capillaries and voids can be utilized to convert forces acting upon the droplets to observable displacements which accurately quantify these forces. The position of droplets within such micro-sensors is governed by surface tension, wettability, and the geometric configuration of the confining walls. If non-wetting fluids are employed, the micro-sensors can also be made to operate as micro-valves, micro-switches, optical micro-shutters, as well as other devices. Besides laving no mechanical parts to wear out, such micro-sensors are inherently immune to many orders-of-magnitude over-actuation. Both circular and non-circular confining structures can be employed.

Actual Paper

As miniaturization of electrical and now mechanical and fluidic systems occurs, the role of physical and chemical parameters has to be recome effects, such as those due to gravity become relegated to minor roles, while other normally inconsequential parameters become elevated in importance and, sometimes become the dominating variables. This "downsizing reappraisal" is vital to successful miniaturization. In a very real sense, new worlds are entered into in which design considerations and forces normally considered negligible in everyday applications become essential to successful utilization of the miniaturized technology.

For example, surface tension and the closely-related phenomena, wettability, are usually not comparable in effect to other physical forces. Surface tension is usually ignored in our macroscopic world when determining fluid flow through pumps and tubes because its effect is many orders-of-magnitude smaller than pressure drop caused by viscosity. This is a direct consequence of the difference in pressure, ΔP , existing between the inside and outside of a spherical droplet, which is given by the Young and Laplace equation of capillary pressure (1-2):

$$\Delta P = 2\gamma/r \quad . \tag{1}$$

In this equal-radii form of the capillary pressure law, γ is surface tension and r is droplet radius. Normally, in most macroscopic fluid flow applications, radii of fluid surfaces are measured in hundreds, if not thousands, of microns. Pressure differences due to surface tension effects are therefore inconsequential, typically measuring far less than atmospheric pressure. By comparison, pressure drops resulting from viscous flow are oftentimes considerably greater than one atmosphere. However, when droplet radius is on the order of microns, pressure drop across the fluid boundary become very large,

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frequently surpassing many atmospheres. This is precisely the reason why fine aerosol droplets are so difficult to form. However, it is not specifically formation of tiny droplets that is the focus of discussion here, but rather their behavior in miniature voids, such as, cavities, capillaries, and channels which are shaped in such a fashion so as to be partially confining to the droplet. The position of droplets within such micro-voids is governed by the surface tension of the droplet fluid, the wettability of the fluid with respect to micro-void walls contacted during displacement, and, the geometric configuration of the walls confining the fluid droplets. Micro-devices fabricated using these micro-voids can be made to operate with wettabilities greater than or less than ninety degrees, but not exactly ninety degrees.

The behavior of such a system is easily understood if one compares it to the mercury intrusion method (3-4) of measuring the pore-size distribution within porous solids. This technique is predicated approach the understanding that the pressure needed to force a non-wetting fluid into a capillary, or pore in a solid, is given by the relationship proposed by Washburn (3):

$$P = 2\gamma \cos\theta/r \tag{2}$$

where θ is the contact angle of the fluid with the material under test, P is the external pressure applied to the non-wetting fluid, and r is the radius of the capillary or pore which is assumed for simplicity to have a constant diameter. This equation is valid for any fluid in contact with a capillary or porous solid having a contact angle greater than ninety degrees. Once the external applied pressure exceeds that needed to force the nonwetting fluid into a constant-diameter capillary or pore, the nonwetting fluid will flow into that particular diameter capillary or pore until it fills it. The volume of the intruded fluid is then a direct measure of that particular capillary's or pore's void volume. If a smaller capillary or pore branches off the larger-diameter void, it will remain unfilled until the external pressure is raised sufficiently high that equation (2) is again satisfied, and the process repeats itself.

In contrast to the mercury intrusion method of determining pore volume, instead of determining the pore volume, the emphasis here is placed appon determining the exact location of the nonfyetting fluid's smallest-radius surface (L_r) as this surface progresses through such an ideal pore system in response to ever increasing external pressure. Although not necessary in actual practice, it is convenient to assume co-axial placement of straight pores in such an ideal system, as shown in Figure 1a. It is also helpful to assume initially that this pore system is under vacuum and that the pores are all circular in cross-section with no branching. When the external pressure applied to the non-wetting fluid is raised, the fluid will fill each successively smaller-diameter circular pore as equation (2) becomes repeatedly satisfied. Clearly, the position of L_r indicates the current external pressure in terms of ranges. If the pressure is between zero and $2\gamma\cos\theta/r_1$, the fluid's smallest-radius surface will appear as shown in Figure 1a. If the pressure is between $2\gamma\cos\theta/r_2$ and $2\gamma\cos\theta/r_3$, this surface will appear as in Figure 1c. And, if it appears as in Figure 1d, all that can be stated is that the pressure is greater than $2\gamma\cos\theta/r_3$. This device is functioning as a rather simple step-function pressure sensor.

However, it does have one specific attribute that most other pressure sensors do not. It can be tremendously over-pressured and still return to its original precision and accuracy repeatedly. As long as the pore walls remain intact and diameters remain unchanged, it will continue to detect pressure ranges accurately because it resets itself to zero each time the external pressure is removed. It then returns to its original sensing capability, which is based solely on surface tension, wettability, and pore geometry.

Determining the location of the non-wetting fluid's smallest-radius surface can be done in a variety of ways including direct optical observation, interfer ometry, electrical continuity or resistance between the two ends of a given pore, and capacitive and/or inductive types of measurements. The initial assumptions of requiring such a pressure sensor to be a straight pore system under vacuum with all cross-sections being circular, with no branching, and with any decrease in diameter only occurring when moving away from the pore entrance can now also be examined. First, the system does not need to be linear and coffaxial. The same type of fluid response will take place in Figure 1 if the pore system is bent into a circle, for example. As long as no reduction in pore diameter takes place during bending and at any point of stepwise diameter change fluid flow remains unimpeded, the system remains equivalent. Second, the system does not have to be under vacuum. If it is not, two options are possible. Either some species of gas can be trapped within the pore structure which then undergoes compression as the fluid's surface L_r advances or the pore structure can be open-ended in which case any enclosed gas is simply expelled as L_r advances. In the first case, L_r will begin moving in a continuous fashion in response to increases in pressure because gas compression provides a back pressure counteracting the applied external pressure. However, this can be calculated and included in sensor design. It gives a pressure sensor much greater operating range as compared to vacuum operation. In the second case the pressure sensor will simply operate in the differential-pressure response mode, and will continue responding in the stepwise manner described initially for vacuum operation with closed pore ends.

The third assumption of requiring that all cross-sections be circular is also not completely necessary. To understand easing of this geometric constraint refer to Figure 2. In this figure pores having a circular cross-section as well as a rectangular cross-section are shown. The minimum dimension of both pores is 2r. The only difference between fluid behavior in the two systems is that complete contact takes place between the fluid and pore wall in the circular pore but not in the rectangular pore. The internal pressure within the fluid, which will equal the external pressure applied in Figure 1, is still given by equation [2] in both cases. The only design consideration that must be taken into account when dealing with a non-circular pore or capillary is that gas flow can take place in corners around the non-wetting fluid when it resides in a non-circular channel. Care must be taken to ensure that external applied pressure does not leak through these corner pathways. This can be achieved either by having a circular cross-section at the entrance to the pore system, by having the pore located in the center of a much larger reservoir wall, or by installing a diaphram over the non-wetting fluid reservoir in such a fashion that it contacts the nonfyetting fluid directly during external pressurization. additional benefit from relaxing the circular cross-section geometric constraint is the creation of new devices. For example, if the ratio of the rectangular larger dimension, w,

to smaller dimension, 2r, is greatly increased and an opaque non-wetting fluid is used, the device will operate like a shutter. When sufficient pressure is applied to form a radius of 2r, the entire width x of the rectangle will fill immediately preventing light from passing through. As soon a pressure drops below that needed to maintain a radius of 2r, the rectangular cavity will empty completely of the opaque non-wetting fluid thereby opening the shutter. Such a shutter will operate mounted vertically, horizontally, or even upside down.

The last two assumption involving ever-decreasing diameters moving away from the pore entrance and lack of branching can also be relaxed with some interesting consequences. Referring to Figure 3, the pore system illustrated now resembles a teardrop. response of the fluids's smallest-radius surface L_r is now continuous rather than stepwise and thereby permits operation as a continuous readout pressure sensor even in the vacuum mode of operation (i.e., without back pressure caused by entrapped gas or openended differential-pressure setup). Figure 4 illustrates several further refinements. For example, two small diameter branch pores that have gas flowing through them are attached to the small end of the teardrop. Also, a form of actuation other than pressure, (in this example a heating coil), is used to move the non-wetting fluid's smallest-radius surface towards the small end of the teardrop. Thermal expansion now makes the system function almost like a thermometer. It is important to recognize that the internal pressure within the fluid is still governed by equation 2. When the heating coil is energized, volume of the non-twetting fluid increases forcing L_r past the arm attached to the side of the device. As long as the arm is smaller in diameter than the radius of the non-wetting droplet at its maximum point of intrusion (i.e., $r_1 < r_2$), gas flow through the arms will cease and none of the non-wetting fluid will enter into the small arms. This device is an on-off valve. The maximum gas pressure that can be controlled is given by equation 2 with the radius being that of the small side arm. If one assumes this radius to be 5 microns and the non-twetting fluid to be mercury with a surface tension of 475 dynes/cm and a contact angle of 130 degrees, the maximum controllable pressure is about 17.7 psi. While this is not very high, it may be quite adequate for many micro-fluidic applications. If the small arm radius drops below 1 micron, the controllable pressure increases to almost 100 psi.

In addition to the use of external pressure and thermal expansion to squeeze non-wetting fluids into miniature shaped cavities, inertial forces or gravity can also be employed quite effectively for the same purpose. Consider the micro-device illustrated in Figure 5. It is very similar in appearance to the pressure sensor shown in Figure 3. Now, however, the mechanism creating internal pressure in the non-wetting fluid is provided by gravity, at least in the static case. Equation 2 still applies, except that pressure is now provided by the hydraulic head of a column of non-wetting fluid of height h and density ρ . Therefore, the following relationship governs displacement of the non-wetting fluid toward the smaller end of the micro-cavity:

$$\rho gh = 2\gamma \cos\theta$$
.

If acceleration of magnitude a is employed rather than gravity as the forcing mechanism, g is replaced by a in equation (3). This micro-device, an accelerometer, would respond to changes in acceleration in a continuous manner. Its accuracy would depend on the rate of decrease in radius as a function of height and the precision of the readout apparatus. It can be positioned in any orientation when functioning as an accelerometer, but will, of course, only measure acceleration in the direction parallel to the cavity. It is again important to notice that if the bottom of the micro-accelerometer cavity is closed off and under vacuum, acceleration many orders of magnitude greater than the designed sensitivity range of the device will cause no problem, assuming walls do not break. It will always return to its initial state when overfload conditions cease, and be just as accurate as before. If it is not closed off, the non-wetting fluid will escape from the micro-device at some acceleration predetermined by system design parameters. As before, in this application non-circular cross-sections can also easily be used.

The foregoing discussion assumed fluids that are non-wetting. This does not have to be the case. If the working fluid wets capillary or pore walls, it is drawn into shaped cavities and pressure is required to expel it. Similar kinds of micro-devices can be designed and constructed using wetting fluids as the enabling technology. One drawback to this kind of micro-fluidic system is that fluid cannot easily be removed from cavity walls causing loss of droplet volume during change of position of fluid surfaces. If the wetting angle is exactly ninety degrees, there is no surface tension effect at all. The fluid essentially does not recognize that it is in a shaped cavity.

Figure 5 is a photograph of shaped cavities that function as a pressure sensor, while Figure 6 shows an accelerometer. These devices were made using microtube fabrication technology (5) that allows virtually any cross-section and axial profile of micro-cavity to be formed. The non-twetting fluid is mercury in both instances.

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